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Publisher's version / la version de l'éditeur:
Proceedings of the 10th International Conference on Aluminum Alloys, 2007, 2006-07-13

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imi 2005-111894-9
CNRC 48835

Laser-Ultrasonic Characterization of the Microstructure of Aluminum

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Keywords: Ultrasound, texture, crystallographic orientation distribution, residual stress, annealing, recrystallization, internal friction, dislocations.

Abstract. Ultrasonic velocity and attenuation measurements are powerful tools to infer much information about the microstructure and properties of aluminum and its alloys. Laser-ultrasonics is a technology that enables doing these measurements remotely, *in-situ* or inline and in a fraction of a second. Therefore, it is possible to characterize the thermomechanical processing of aluminum alloys with unprecedented time resolution. This paper reviews the physical principles that allow relating velocity and attenuation measurements to various materials properties and microstructural features such as elastic moduli, crystallographic distribution orientation (texture), residual stresses, recrystallization and dislocations. *In-situ* (in laboratory furnaces) and in-line measurement examples from the Industrial Materials Institute research group are reviewed and presented.

Introduction

Ultrasonics is a probe of choice for inspecting the interior of materials because ultrasound can penetrate deeply. For the purpose of detecting defects inside metals, it is the most practical alternative to heavy doses of x-rays or gamma rays. Ultrasonics can also be used to infer much information about the bulk microstructure of metals. Unlike x-rays or neutrons diffraction, the obtained information is not as detailed but it can be obtained much faster: the measurements last a few microseconds and can be repeated up to thousands of times per second. Also, the information can often be obtained without sample preparation, off-line or in-line during processing.

The last two decades have seen the emergence of new laser-ultrasound technology that allows generating and detecting ultrasound with pulsed laser radiation. Laser-ultrasonics offers many advantages over conventional ultrasonics utilizing contact piezoelectric transducers. First, it is a non-contact technique capable of working at distances ranging from 1 cm to 1 meter or more. This allows doing measurements at any temperature, under any processing atmosphere, and on moving parts. The only requirement is that there be a direct line of sight to the part. There is no need for an acoustic bond between some transducer and the part and thus there is no interaction (ringing, internal echoes, etc...) between the transducer, the bond, and the part. This greatly facilitates the analysis and greatly improves the accuracy and reliability of the results. Laser-ultrasonics is also a wideband and multimode technique. It can measure simultaneously compressive or shear acoustic waves, surface waves or other waves, and it can do so over a frequency bandwidth of 1-100 MHz or more, providing in a single measurement much more information than what a single piezoelectric transducer could ever provide. These advantages have enabled us to advance the ultrasonic measurement of the microstructure of metals in general, and of aluminum in particular. This paper is a short review of work done in our laboratories.

Ultrasound Velocity, Texture, Residual Stresses

Generally the elastic properties of single crystals differ in various directions. Consequently, the ultrasound velocity also depends on propagation direction [1]. Metals, however, are polycrystalline

aggregates. Their average bulk aggregate properties depend both on single crystal properties and on their volumic average over all possible single-crystal orientations, weighted by the probability of finding these orientations [2]. Clearly, more strongly textured aggregates have more anisotropic properties. Also, the higher is the anisotropy of the single crystal, the higher is the anisotropy of the aggregate. The aluminum single crystal is weakly anisotropic. As a result, the elastic anisotropy of aluminum and its alloys tends to be small. Nevertheless, ultrasonics can measure such anisotropies easily.

For aggregates of cubic single crystals (such as Al or Fe) having an orthorhombic aggregate symmetry (the case of lowest symmetry that is of practical significance), in the approximation of small anisotropy of the aggregate (a valid approximation because the Al single crystal itself is weakly anisotropic), the sound velocity in any of the symmetry planes can be expressed in a simple expression involving single crystal elastic constants, texture coefficients (expansion coefficients of the ODF, i.e. of the orientation distribution function), and propagation direction. For example, the velocity of bulk compressive waves in the plane of a metallic sheet or plate is given by:

$$V_P = V_{P0}\left(1 - 0.228 W_{400} + 0.481 W_{420}\cos 2(\theta - \theta_W) - 0.636 W_{440}\cos 4(\theta - \theta_W)\right), \quad (1)$$

where V_p is the sound velocity (in m/s) of the pressure waves, V_{p0} is the velocity in a sheet that would have isotropic texture, W_{4m0} are the texture coefficients in a coordinate system where the z axis is normal to the plane of the sheet, θ_W is a texture symmetry direction in the plane of the sheet (e.g. the rolling direction) and can often be set equal to zero. The numerical factors are calculated from single crystal elastic constants. Numerous authors have utilized similar expressions to infer the texture coefficients of aluminum sheets. These coefficients have also been correlated with plastic deformation properties relevant to formability, such as the plastic strain ratio.

Other materials properties can affect the sound velocity. The single crystal elastic constants also depend on temperature, stress, alloying elements, and dislocation structures. Usually, for a specific metallic alloy, texture is the main factor affecting velocity. However, as mentioned above, the single crystal elastic constants of Al are weakly anisotropic. Moreover, Al has relatively high acousto-elastic coefficients, i.e. the dependence of its elastic constants on stress is larger than for many other metals. Neglecting texture, the in-plane dependence of the acoustic velocity on applied stress is

$$V_P = V_{P0}\left(1 - 4.35\times10^{-5} T_m + 6.0\times10^{-5} T_d \cos 2(\theta - \theta_{RS})\right), \quad (2)$$

where T_m is the mean applied stress measured in MPa, T_d is half the difference between the two principal stresses, and θ_{RS} is one of the principal directions and can often be set equal to zero. The numerical factors are calculated from single crystal elastic third order elastic constants in the case of isotropic texture symmetry. They can also be measured empirically. Such equations are the basis for ultrasonic measurement of residual stresses. In most cases, one only attempts to measure T_m. Texture effects generally dominate residual stress effects, i.e. variations of W_{4m0} usually produce larger velocity changes than variations of T_m. Therefore, in practice, one measures the velocity difference between a stressed sample and a reference sample of the same alloy and texture.

When measuring residual stresses with ultrasound, a problem arises when the process utilized to create the stress also changes texture. Such texture changes probably occur with all surface processing techniques such as shot peening, laser peening, or low plasticity burnishing (LPB). To solve this problem and to extend the applicability of ultrasonics to the measurement of T_d and θ_{RS}, we have recently devised a new measurement method [3].

The ultrasound velocity of two types of surface acoustic waves, Rayleigh waves and surface skimming compression waves, is measured as a function of θ, both on the surface in its original state, and after surface processing. By writing the appropriate equations (Eqs. 1 and 2 of this paper

and similar equations with different numerical factors for the Rayleigh waves) and solving the inverse problem, it has been possible to separate the effects of changes in texture and residual stresses. This has allowed measuring T_m and T_d, and θ_{RS} independently of texture changes caused by applying by the LPB surface processing technique. Note that in this particular case, the LPB process was intentionally adjusted to produce strongly anisotropic residual stress (i.e. high value of T_d).

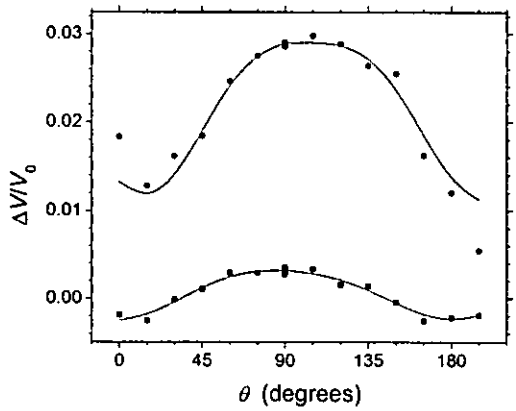

Figure 1: Bottom square symbols and line: Angular variation of $\Delta V_R/V_{R0}$ in the 20-60 MHz frequency bandwidth. Top circular symbols and line: Angular variation of $\Delta V_P/V_{P0}$ in the 3-12 MHz frequency bandwidth. The symbols are experimental data and the lines pertain to the fitted model.

Fig. 1 illustrates the measurement. The velocities of the two surface waves were measured as a function of propagation direction on a 1-inch thick plate of AA7075. The velocity differences between the processed and the as-received surfaces, ΔV_R and ΔV_P, was calculated and normalized by the absolute velocities in the zero degree direction, V_{R0} and V_{P0}, for the Rayleigh and surface skimming pressure waves, respectively. These were fitted to a linearized equation equivalent to

$$\left(V - V_0\right)/V_0 = A + B\cos 2(\theta - \theta_2) + C\cos 4(\theta - \theta_4), \tag{3}$$

where A, B, and C are fitted amplitudes and θ_2 and θ_4 are the fitted symmetry direction of the velocity measurements. Clearly, Fig. 1 shows that these symmetry directions are not the same for the two types of waves. This is because θ_{RS} and θ_W are not necessarily the same and because the different sensitivities of the two ultrasonic waves on texture and residual stresses.

The inverse problem was solved and values of T_m and T_d, and θ_{RS} were obtained. Table 1 compares the ultrasonic measurements to surface x-ray measurements, to hole drilling measurements, and to a second set of x-ray measurements as a function of depth. The ultrasonic measurement averages the properties over *some* depth. For comparison purpose T_m for the two measurements as a function of depth were averaged between 0 to 500 μm. Below 500 μm, they drop rapidly to small values. Table 1 shows that the ultrasonic measurement yields a reasonable value of T_m and it overestimates somewhat T_d.

To complete this study, there remains to analyze changes in texture with depth and compare x-ray and ultrasonic measurements. In the longer term, it is hoped that the frequency dependence of the ultrasonic measurement can be used to obtain a complete depth profile.

Table 1: Comparison of ultrasonic, x-ray, and hole drilling measurements of residual stresses.

	T_m [MPa]	T_d [MPa]	θ_{RS}
Ultrasonics	−498	−143	12°
Surface x-rays	−305	−118	n/a
Hole drilling averaged over depth	−463	From −110 to −10	~9°
X-rays averaged over depth	−510	From −115 to −15	~0°

Recrystallization

During the recrystallization of deformed microstructures, there usually occurs a texture change. Therefore, recrystallization can be sensed by measuring ultrasound velocity changes, from an initial value to the recrystallized values. Fig. 2 illustrates this principle during isothermal annealing at 350 °C of a sample AA6111, cold-rolled by 60% [4]. On the left, the fractional change of the W_{400} texture coefficient measured using neutron diffraction is compared to the fractional change in compression wave velocity in the sheet thickness which also depends principally on W_{400}. These fractional changes are assumed to be proportional to the recrystallized fraction, as indicated by the y-axis label. The right of Fig. 2 compares three measurements of the recrystallized fraction during the isothermal annealing of a 40% cold-rolled AA5754 sample. The three measurements are derived from the ultrasound velocity in the thickness of the sheet, from metallography, and from a yield stress measurement. The three measurements agree quite well. However, the *in-situ* ultrasonic approach allows obtaining much more detailed information in a single experiment. Using this measurement technique, in-line measurements of recrystallization were made successfully at Commonwealth Aluminum Corporation, in Carson, CA [5].

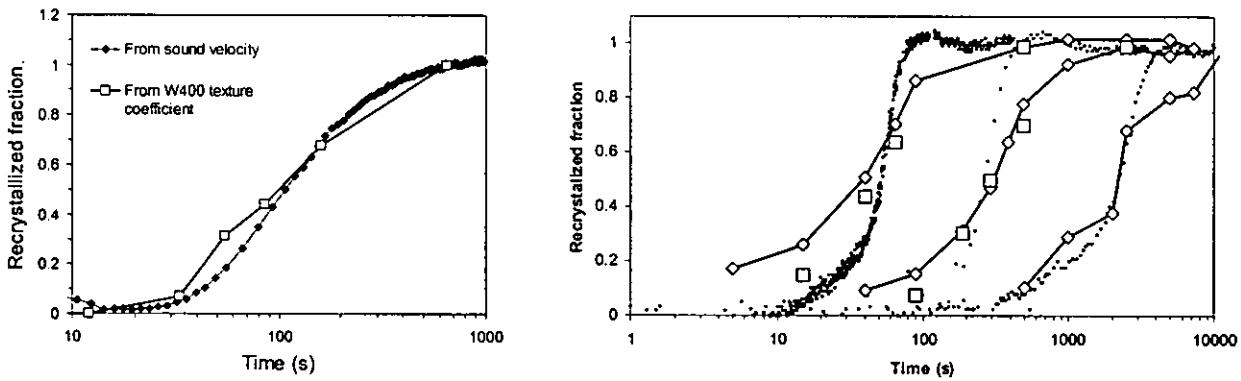

Figure 2: Left: Recrystallized fraction (fractional change in W_{400}) during isothermal annealing of a 60% cold-rolled AA6111 sample. Right: Recrystallized fraction during isothermal annealing a 40% cold-rolled AA5754 samples. The solid circles are from ultrasonics, the connected open diamonds are from yield strength, and the open squares are from metallography.

Attenuation and Internal Friction

In most metals, the main contribution to the attenuation of a plane propagating ultrasonic wave is scattering by grains. In a simple model, the wave is traveling in an effective continuous medium. When it encounters a grain, the elastic properties of that grain may differ from the effective properties of the continuous medium because, as discussed above, the elastic properties depend on grain orientation and because the grains are oriented more or less randomly. This change in local elastic properties causes scattering of the acoustic plane wave, thus resulting in the attenuation of the plane wave. If the grains are more anisotropic than the scattering mechanism is more effective. Because aluminum has a rather low anisotropy, the scattering mechanism does not work well in aluminum. For comparison, grain scattering is about 25 to 30 less effective in aluminum than in iron. Instead ultrasonic attenuation tends to be dominated by other mechanisms.

The ultrasonic attenuation of compression waves in the frequency range of 30-200 MHz for AA5754 samples is illustrated in Fig. 3. With the plane wave experimental configuration utilized, ultrasonic diffraction and beam spreading are negligible. The symbols are measurements on the following samples: H1: as-received from a hot band (from a hot rolling mill, requires annealing to recrystallize); D3: H1 material annealed 30 minutes at 600 °F (mostly recovery, little recrystallization); 90: fully annealed; G4: H1 material annealed 4 hours at 950 °F (fully recrystallized with grain growth) [6]. The straight lines through some data sets are linear least

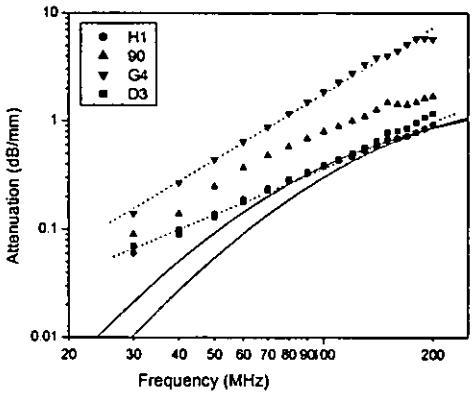

Figure 3: Attenuation as a function of frequency in AA5754. The dotted lines are empirical power law fits to sample H1 and G4. The solid lines are scattering model predictions using the measured grain size of sample G4 (top line) and 90 (bottom line).

squares fits to a power law relationship. Theoretically, in the limit of low frequencies, grain scattering should cause attenuation to be proportional to f^n, with $n = 4$, and where f is frequency. As frequency increases, the calculated n decreases to less than 2. Model predictions based on a 14.4 and 19.5 μm grain size (i.e. the grain size of samples 90 and G4) are shown as solid lines in Fig. 3. This model worked very well for fully recrystallized low carbon steel grades [7] but here, it underestimates the total attenuation by nearly a factor of 10. Moreover, the measured n varies from 1.4 (Sample H1) to 2.0 (sample G4) and does not decrease with frequency (except for sample 90). Such behavior is not indicative of grain scattering but rather of absorption (or internal friction).

To study absorption independently of attenuation, an ultrasonic reverberant technique [8,9] was developed. A small sample is held ultrasonically decoupled from its environment. A single laser pulse generates a wide spectrum of acoustic frequencies in the sample. The laser interferometer monitors the decay of the reverberating ultrasound. A time-frequency analysis of the detected signal allows calculating the decay time (or absorption rate) as a function of frequency. In this technique, most of the ultrasonic energy is in the shear modes of vibration. Therefore, the results cannot be quantitatively compared to those of Fig. 3 for compression waves.

Fig. 4 shows the internal friction ($1/\pi$ times the natural logarithm of the amplitude loss per acoustic period) measured with the reverberant technique in a sample of cold rolled AA5754 during heating from room temperature to 500 °C (measurements were also made during cooling). The left graph shows that the high temperature dependence follows an Arrhenius law with activation energy of about 1.0 eV. It also displays a sharp feature occurring between 356 and 376 °C, the temperature at which the sample is expected to recrystallize [10]. The right graph shows that internal friction is proportion to f^m at the higher frequencies, where m goes from positive at low temperature, to negative at high temperature. The right graph also highlights the high increase in internal friction at high temperature and low frequency.

Further data analysis indicates that the high-temperature, low-frequency limit of the data (during cooling when microstructural modification should be completed) is well described by Schoeck's model which is based on a broad spectrum of relaxation mechanisms (not shown).

Most authors attribute the low temperature internal friction to the bowing of pinned dislocations between their pinning centers, as described by the Granato-Lücke relaxation model. This model predicts that the internal friction increases with dislocation density, dislocation loop length and, in the frequency range of interest here, as f^m where $0 \le m \le 1$. This model is consistent with Fig. 3 where absorption increases as frequency to the power $n = 2$ or less ($m = n$-1). It is also consistent with Fig. 4 at low temperatures. When temperature is raised, m lessens. This is probably caused by the recovery of dislocations which begins near 150 °C (0.0024 K^{-1}). Near 333 °C (0.0016 K^{-1}), the slope is nearly zero and soon after recrystallization occurs and there are very few dislocations left.

At higher temperatures, the low-frequency high temperature background described by Schoeck's model dominates. These observations in the 10^7-10^8 Hz range are compatible with what has been observed in the 10^{-4}-10^2 Hz range by standard internal friction techniques [11,12]. It is hoped that further such studies will allow the development of laser-ultrasonic techniques capable of extracting the dislocation contribution to the ultrasonic signals.

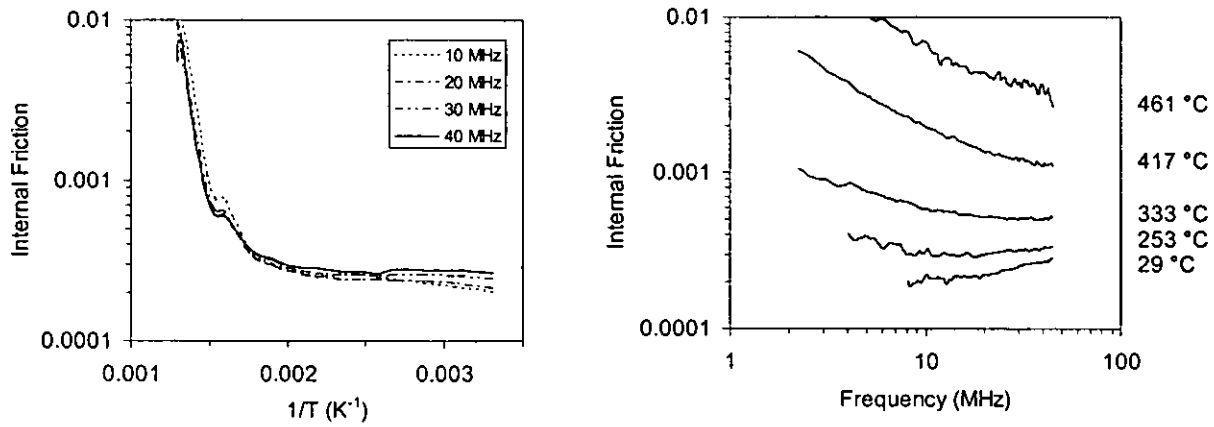

Figure 4: High temperature absorption during heating a sample of cold-rolled AA5754.

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Title: Laser-ultrasonic characterization of the microstructure of aluminum

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Abstract

Ultrasonic velocity and attenuation measurements are powerful tools to infer much information about the microstructure and properties of aluminum and its alloys. Laser-ultrasonics is a technology that enables doing these measurements remotely, *in situ* or inline and in a fraction of a second. It is therefore possible to characterize the thermomechanical processing of aluminum alloys with unprecedented time resolution. This presentation will review laser-ultrasonic technology and the physical principles that allow relating measurements of velocity and attenuation as a function of frequency, polarization, and propagation direction to various materials properties and microstructural features such as elastic moduli, crystallographic distribution orientation (texture), residual stresses, and grain size. *In situ* (in laboratory furnaces) and inline measurement examples from the Industrial Materials Institute research group will be presented.